United States
Environmental Protection Agency
Office of Transportation and Air Quality
National Vehicle and Fuel Emissions Laboratory
2565 Plymouth Road
Ann Arbor, MI 48105

Gas Naming Procedure

This procedure is written for the Environmental Protection Agency, National Vehicle and Fuel Emissions Laboratory (NVFEL) internal use. The use of specific brand names by NVFEL in this procedure are for reference only and are not an endorsement of those products. This document may be used for guidance by other laboratories.

NVFEL Reference Number

105B

Implementation Approval

Original Test Procedure Authorized on 11-28-79

Revision Description

(1)	09-30-1994	This procedure has been edited as described in EPCN #147. Page layout, grammatical, and spelling changes have been made, but there were no technical revisions to the procedure.

Table of Contents

Attachments

1. Purpose

The purpose of this procedure is to determine the concentration of a gas mixture by comparing it to a series of known standard gases such as National Bureau of Standards (NBS) bottles, Environmental Protection Agency (EPA) gravimetrics, and EPA secondaries.

All standard cylinders of gases used in vehicle certification must first have undergone the correlation procedure (TP 403, Standard Gas Correlation).

2. Test Article Description

A gas blend or mixture of two or more gases under pressure in a cylinder whose concentration must be determined

3. References

3.1 Instruction manuals for the instruments on the Master Analysis System

3.2 EPA Laboratory Safety Manual

3.3 “Federal Register” Vol. 43, No. 24, June 28, 1977, Section 86.114

3.4 “Proceedings of the EPA/Industry Quality Control Symposium on Gas Standards - Management and Traceability Practices,” July 27, 1977, particularly the “Quality Assurance Paper on Calibration Gas Management,” by Don Paulsell, March, 1976

3.5 LNS User's Manual

4. Required Equipment

4.1 The following components contained on the Master Analysis System A251:

4.1.1 Beckman Model 400 Flame Ionization Detector for measuring hydrocarbons

4.1.2 Horiba Model AIA-23A Infrared Analyzers with different cell lengths for analyzing CO and CO_2

4.1.3 Beckman Model 951 NO/NOx Analyzer

4.1.4 Gow-Mac Series 550 Thermal Conductivity Gas Chromatograph for analyzing H_2He and H_2/N_2

4.1.5 Bendix 8205 Methane Analyzer

4.1.6 Taylor-Servomex Type OA.137 Oxygen Analyzer

4.1.8 Fluke 8000A Digital Multimeter

4.1.9 Hewlett-Packard 7132A strip chart recorder, used when naming methane

4.2 Regulators and sample lines. Sample lines should be Teflon covered with braided stainless steel, with stainless steel fittings.

All regulators must be dual stage. NOx regulators must be stainless steel, connection size CGA 660 with Teflon gaskets.

Brass regulators are used for all other gases, connection size CGA 590 for air, CGA 580 for nitrogen, and CGA 350 for all other gases.

4.3 Standard gases of varied concentrations: NBS cylinders, EPA gravimetrics, and EPA secondary cylinders.

All standard gases must first be subjected to the correlation procedure (TP 403) before they may be used for naming gases, with the exception of FID fuel standards which are not correlated at the present time.

4.4 Zero Gases - Hydrocarbon-free air or 99.9% nitrogen as required

4.5 Form LB105, Gas Analysis Data Worksheet

4.6 Form AA-601, Exhaust Gas Analysis Calibration Data Form

5. Precautions

5.1 Technicians must be familiar with the EPA Laboratory Safety Manual sections on cylinder safety and the safe handling of test gases.

5.2 All cylinders and equipment (regulators, lines, instruments, etc.) must be checked for damage, leakage, and cleanliness.

5.3 Labels on all cylinders to be used must be carefully read to determine contents.

6. Visual Inspection

All visual inspections are included as part of the test preparation and test procedure.

7. Test Article Preparation

7.1 Select at least six standard cylinders, eight when naming CO and CO_2, with sequential concentrations of the gas type to be tested.

The highest standard concentration should be slightly higher than the estimated sample concentration and the lowest standard concentration should not fall below 10% of the full range reading (e.g., if the estimated concentration of the sample cylinder is 950 ppm, the highest standard bottle should approximate 1,000 ppm and the lowest should approximate 100 ppm).

The concentrations of the remaining standard cylinders should be spaced at nominal intervals of 10-15% to insure an even spread of data points in the curve.

7.2 The ambient room and cylinder temperatures must be between 68-86 °F while the analysis is run. Be sure to allow at least 24 hours for cylinders that have just been received to adjust to this temperature range.

7.3 Make sure the cylinders are properly secured. Attach suitable pressure regulators to the sample and standard cylinders.

Check for leaks with leak detector solution.

7.4 Analytical instruments used for test gas calibrations should always remain in a standby mode to insure maximum accuracy and stability.

If the instrument has been shut off, it must be warmed up according to the analyzer operations manual.

7.5 Set the instrument at the proper pressure and check the flow rate.

8. Test Procedure

All calibration points must be stable readings taken from the DVM. A stable reading must be determined by the operator as that reading with as little fluctuation as possible. The operator must allow sufficient time for the reading to stabilize.

If a reading fluctuates between two points, the operator estimates the average reading (e.g., 90.1-90.2 = 90.15). Fluctuations greater than .2 units should be noted on LB105.

If methane is being analyzed, the strip chart is used to verify instrument stability. Note on the strip chart all corresponding readings taken from the DVM.

100 Calibrate Analyzer

101 Zero:

Switch the analyze to zero and allow zero gas to flow through it. Adjust the pressure if necessary, check the flow rate, and select the proper range. These must remain constant during the analysis.

Adjust the zero potentiometer to read between ± 0.1 unit of zero.

102 Span:

Attach the analyzer input line to the highest concentration standard gas to be used. Switch the analyzer to span, wait for the reading to stabilize, and adjust the span potentiometer so that the reading falls between 92 and 99 percent of full scale.

103 Zero:

Switch the analyzer to zero and wait for the reading to stabilize.

If the reading does not fall within ± 0.1 unit of zero, adjust the zero potentiometer and repeat the span-zero until the zero point remains within tolerance.

200 Analyze Standard Cylinders

201 Switch the analyzer to span. Allow the reading to stabilize and record it on LB105.

202 Disconnect the input line from the span gas and connect it to the next lower concentration standard cylinder.

Record the DVM reading after it stabilizes. Repeat this process for the remaining standard gases in order of highest concentration to lowest.

203 After the lowest concentration standard is recorded, reconnect the highest concentration standard and note the DVM reading after it stabilizes.

If the reading is not within ± 0.1 unit of the first span reading, zero the analyzer and repeat Step 200 until the two span points agree.

204 Switch the analyzer to zero and obtain a stable reading.

If it is not within ± 0.1 unit of zero, calibrate the analyzer, and analyze the standard gases again (Steps 100-200).

300 Analyze Sample Gas(es)

301 Disconnect the input line from the span bottle and connect it to the sample bottle. Switch the analyzer on span and record the DVM reading after it stabilizes.

302 If more than one sample is to be analyzed on the same curve, run the sample bottles in order of highest nominal concentration to lowest.

303 Repeat the analyzer calibration (Step 100). If either the zero or span point has drifted out of tolerance, adjust and repeat Steps 100, 200, and 300.

9. Data Input

9.1 The operator completes Form LB105 during the procedure, giving the Analyzer ID, usage code, date, who the analysis is for, the type of gas and diluent, range, pressure, and flow rates.

The cylinders are listed by number, nominal or known concentration, and by the digital voltmeter reading. Four columns are provided for the DVM readings in the event of rerun curves.

9.2 The operator completes Form AA-601 using the information recorded on Form LB105.

9.2.1 Lines 1-3, “Instrument Identification,” are completed using the codes given on the back of the form.

9.2.2 Line 4, “Limits,” is completed. The deflection limits define the upper and lower limits of valid deflection readings on the DVM.

The range change limits define the upper and lower deflection readings on the DVM that signal the need for a range change to computers on real-time systems.

9.2.3 Line 5, “Operator's Comments,” identifies the recipient of the results (e.g. EPA, Hamilton, Scott, etc.) and the type of standards used (gravimetric, NBS, secondaries).

9.2.4 Columns 1-11 of Line 7 are completed using the codes on the back of the form as follows:

Columns 1-2, “zero-span type” - always 01 (“no software zero and span”)

Columns 4-5, “curve form” - “01” if the curve is to be forced through zero; “02” if the curve is to have a finite intercept.

Column 8, “degree of fit” - “2” if the chemiluminescence, FID, gas chromatograph, methane, or oxygen analyzer was used; “4” if the NDIR analyzer was used.

Column 11, “weight factor” - always “2,” which minimizes percent of point deviations.

Columns 16-51 are applicable only if a blender was used.

9.2.5 Lines 8-9 are applicable only if a blender was used.

9.2.6 Lines 10-29 are concerned with the cylinders involved in the analysis:

Columns 1-12 - the cylinder numbers are listed.

Column 14 is applicable only if a blender was used.

Column 16 - “X” if the cylinder concentration is “known.”

Column 18 - “X” if the cylinder concentration is “to be named.”

Columns 20-32 are applicable only if a blender was used.

Columns 34-44 - the known or nominal concentration is listed for each cylinder.

Column 46 - “X” if the cylinder is to be used as a calibration data point. All such cylinders must have a known concentration value.

Columns 48-55 - the DVM readings taken from Form LB105 are recorded to two decimal places.

10. Data Handling

10.1 A Data Processing Request Form is completed, and Form AA-601 is turned in with it for processing.

10.2 The processed data sheet "Analyzer Calibration Curve Analysis" is obtained from Computer Operations.

11. Data Review and Validation

11.1 The technician examines the Analyzer Calibration Curve Analysis and determines the validity of the curve.

11.1.1 A valid curve is determined by noting the figures in the column under "Curve Fit Deviation" marked "% point."

If NBS or EPA secondary cylinders are to be named, the figures in this column must be within ± 0.5% for the curve to be valid.

If working or vendor's cylinders are to be named, the figures in this column must be within ± 1.0% for the curve to be valid.

11.1.2 No inflection points are allowed in the curve. These are flagged in the printout, and indicate problems either with the analyzer or with the gas.

11.1.3 The percent of non-linearity routinely found under the data table of the printout must be less than 10. Non-linearity greater than 10 is flagged in the Quality Control Comments of the printout.

In such cases, the Section Chief should be notified for further action.

11.1.4 The plus and minus signs of deviations (±) must be randomly distributed throughout the curve.

If three or more of the same sign appear clustered at the center or endpoint, the Section Chief should be notified for further action.

If the curve is valid, locate the sample "name" on the "Analyzer Calibration Curve Analysis" in the section called "Cylinder to be Named." The figure under the column called "Curve Fit Calc. Conc. (CFC)" is the "name" of the sample gas.

If the curve is not valid, the input data must be checked for accuracy, corrected, and reprocessed if necessary.

If the input data is found to be accurate, but the curve is invalid, a problem with the cylinder contents or analyzer is indicated. The curve must be rerun to verify this. Once the problem is verified, the source must be determined and rectified.

If the problem is with inaccurate standard cylinders, these may be removed from the curve by removing the appropriate x's in Columns 16 and 46 of Form AA-601, adding an "X" in Column 18, and reprocessing the data. The abnormal cylinders should either be blended again or returned to the vendor.

12. Acceptance Criteria

12.1 All zero and high span points must fall within ±0.1 unit of the original readings.

12.2 The curve must be valid according to the criteria in Step 11.1 before the sample gas name is accepted.

13. Quality Control

13.1 All analytical instruments must be properly warmed up prior to use.

13.2 The type of standards used as calibration data points must be chosen according to the function of the bottles to be named:

13.2.1 All in-house primary standards (NBS and gravimetrics) may undergo the naming procedure as a check for accuracy. However, the names derived from this procedure are never used as known concentrations for these standards.

When the primary standards are undergoing the naming procedure, other gravimetrics should be used as data points in the curve.

13.2.2 When EPA secondary standards are being named by this procedure, primary standards should be used as data points in the curve.

The name derived from this procedure serves only as a check against the calculated concentration obtained from TP 403.

13.2.3 When EPA working cylinders are being "named" by this procedure, EPA secondary standards should be used as data points in the curve.

The name derived from this procedure is accepted as the actual concentration and the cylinder is accordingly labeled (see Step 14.2 below).

14. Documentation

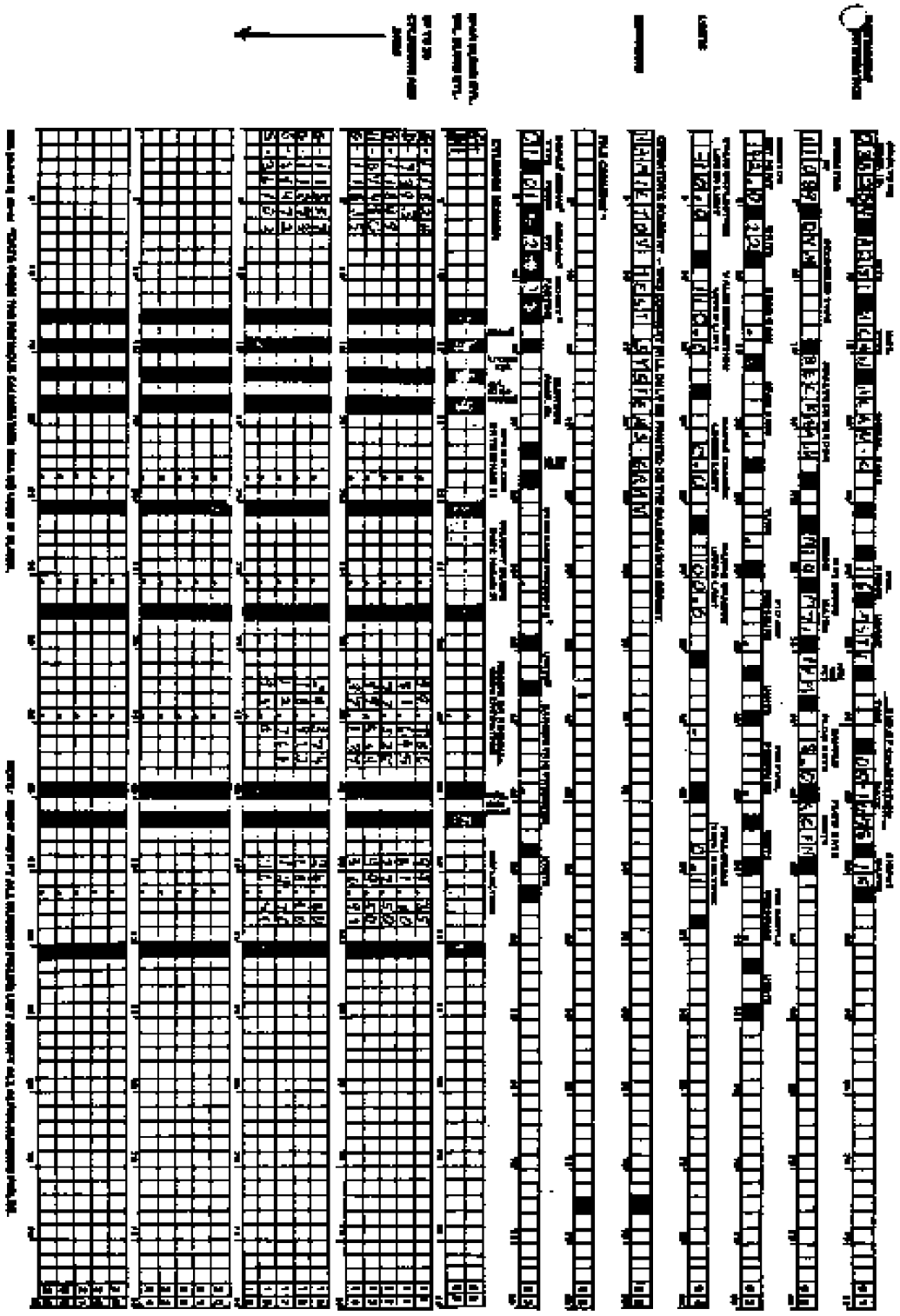
14.1 Forms LB105, AA-601, and the Analyzer Calibration Curve Analysis are filed separately in the Master Analysis Room.

14.1.1 Forms LB105 and AA-601 are filed chronologically according to the gas type. Methane strip charts are attached to and filed with Form LB105.

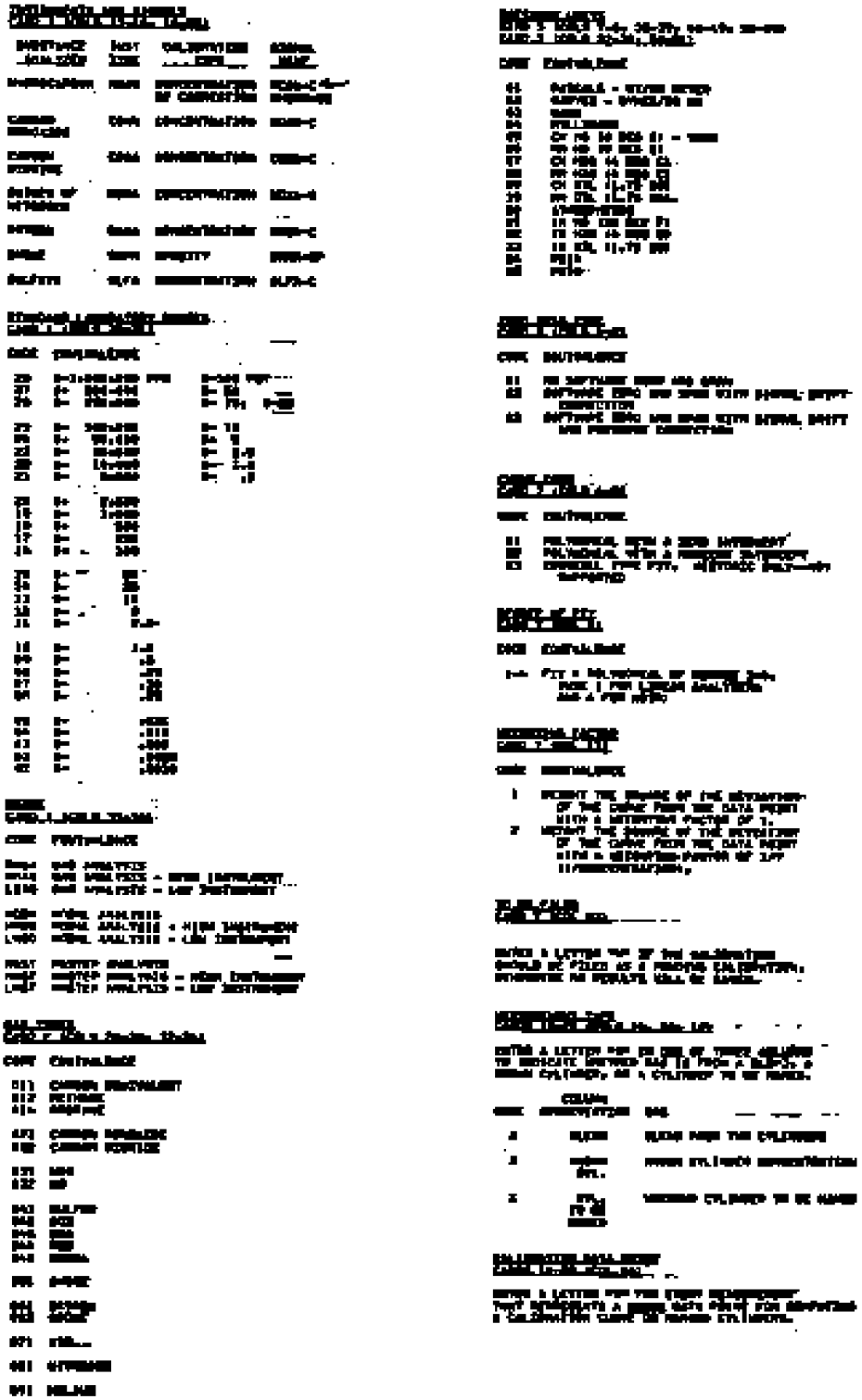
14.1.2 The Analyzer Calibration Curve Analysis printouts are filed chronologically, according to gas type, in individual notebooks for NBS and gravimetrics, secondaries, working, and non-EPA cylinders.

14.2 A blue sticker tape label is affixed to each EPA working cylinder giving the cylinder number, the gas blend, and the “Curve Fit Calc. Conc.” taken from the Analyzer Calibration Curve Analysis in either ppm or percent.

Attachment A



Attachment B



Attachment C

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**** PROCESSED: 13:01:16  05-17-78

*************************************************
*************************************************
***                                           ***
*** ANALYZER CALIBRATION CURVE ANALYSIS       ***
***                                           ***
*************************************************
*************************************************

 AAA      22222    5555555
AAAAA    2222222   5555555     1
AA  AA       22    55          1 11
AAAAAAA     222    555555        11
AAAAAAA   222          55        11
AA  AA   2222222   5555555       11
AA  AA   2222222    55555      11111
```

END CALIB AT TIME	08 8	ANALYZER VENDOR	BECKMAN	SAMPLE FLOW RATE	0.0 SCFH	VALID DEFL. UPPER LIMIT	110.000
CALIBRATION DATA	5-16-78	INSTRUMENT NAME	BEAM	MONITOR SET POINT	42.0 IN H2O	VALID DEFL. LOWER LIMIT	-10.000
TEST SITE NUMBER	A851	EPA IN CAL ID NO	036884	ZERO GAIN SETTING	0.0	RANGE CHANGE UPPER LIMIT	100.000
GAS ANALYZED	PROPANE	SIGNAL LEAD	INFRAM-C	SPAN GAIN SETTING	0.0	RANGE CHANGE LOWER LIMIT	0.0
DILUENT GAS	NITROGEN	HARDWARE RANGE		TUNE READING	0.0	FULL-SCALE (100%) DEFL.	100.000
CONCENTRATION UNITS	PPM	USAGE	NBT1	FID AIR PRESSURE	0.0	FULL-SCALE (100%) VOLTAGE	0.100
STANDARD LAB RANGE	16	CALIB GAS SOURCE		FID FUEL PRESSURE	0.0	FULL-SCALE (100%) CONC.	100.0
OPERATOR ID NO	11089	BLENDER DECAL ID		FID SAMP PRESSURE	0.0	RECORDER TYPE	IDMM

FILE COMMENT :

OPERATOR COMMENT : HAMILTON TEST SYSTEM--GRAY.

CYLINDER NUMBER	BLENDER METER READINGS SPAN	BLENDER METER READINGS DILUENT	BLEND RATIO	CONCENTRATIONS CYLINDER (BLENDED)	CONCENTRATIONS CALCULATED	ANALYZER SIGNAL MEASURED (% A)	ANALYZER SIGNAL CORRECTED (% C)	CALIBRATION DATA POINT	CURVE FIT DEVIATIONS % POINT	CURVE FIT DEVIATIONS % FULL-SCALE	CURVE FIT DEVIATIONS % FROM LAST CALIBRATION
	0			0.0							
		5		0.0					-0.008	-0.039	0.0
				94.79	94.39	94.350	94.350	K	-0.047	-0.079	0.0
G-11029	0.0	0.0	0.0	81.44	81.51	81.500	81.500	K	0.123	0.095	0.0
D-7393	0.0	0.0	0.0	77.42	77.42	77.550	77.550	X	0.053	0.026	0.0
G-11449	0.0	0.0	0.0	64.45	49.58	44.500	45.500	X	-0.112	-0.116	0.0
M-49-89	0.0	0.0	0.0	37.14	37.02	36.950	36.450	K	0.131	0.081	0.0
G-13103	0.0	0.0	0.0	24.37	24.45	24.400	24.400	K	0.172	0.023	0.0
R-11457	0.0	0.0	0.0	18.92	18.46	18.900	18.900	X	-0.249	-0.032	0.0
R-11073	0.0	0.0	0.0	12.76	12.78	12.700	12.700	X	0.0	0.0	0.0
D-49-72	0.0	0.0	0.0	95.40	95.4	95.500	95.500				
5-36102	0.0	0.0	0.0								
								AVERAGE DEVIATION	0.101	0.067	0.0

NONLINEARITY = -0.3 PERCENT

ANALYSIS OF CYLINDERS TO BE NAMED

CYLINDER NUMBER	NOMINAL CONCENTRATION (NC)	LINEAR FIT SIGNAL LOW	LINEAR FIT SIGNAL HIGH	LINEAR FIT SIGNAL CYLINDER	LINEAR FIT CALC. CONC. (LC)	CURVE FIT CALC. CONC. (CC)	CONCENTRATION RATIOS NC/CC	CONCENTRATION RATIOS LC/CC
5-36102	95.0000	81.500	94.350	95.500	95.5534	95.5416	0.99463	1.00010

Attachment C Continued

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**** PROCESSED: 13:48:16  05-17-78

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***                                           ***
*** ANALYZER CALIBRATION CURVE ANALYSIS       ***
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```
END CALIB AT TIME  :   01 0      ANALYZER VENDOR      :BECKMAN    ZERO SPAN TYPE    :   1
CALIBRATION DATE   : 5-16-78     INSTRUMENT NAME      :  HCAN     CURVE FORM        :   1
TEST SITE NUMBER   :  4251       EPA DECAL ID NO      : 03R040    DEGREE FIT        :   3
GAS ANALYZED       :PROPANE      SIGNAL LEAD          :HCAN-C     WEIGHTING FACTOR  :   2
FULL-SCALE CONC    :    100.0    HARDWARE RANGE       :
CONCENTRATION UNITS:  PPM        USAGE                :  MST1
STANDARD LAB RANGE :  16         OPERATOR ID NO       : 11049
```

EQUATIONS AND COEFFICIENTS

$X_C = X_N$

$A5*X_C^4 + A4*X_C^3 + A3*X_C^2 + A2*X_C + A1$ = PPM PROPANE/NITROGEN

A1 = 0.0
A2 = 0.10028662E+01
A3 = -0.25238695E-04
A4 = 0.0
A5 = 0.0

CALIBRATION OFFSET = 0.0

CALIBRATION TABLE — PERCENT FULL-SCALE CHART DEFLECTION VS PPM PROPANE/NITROGEN

0.	0.0																		
1.	1.08	11.	11.03	21.	21.05	31.	31.06	41.	41.08	51.	51.04	61.	61.08	71.	71.04	81.	81.07	91.	91.05
2.	2.01	12.	12.05	22.	22.05	32.	32.07	42.	42.08	52.	52.08	62.	62.08	72.	72.08	82.	82.07	92.	92.08
3.	3.03	13.	13.03	23.	23.05	33.	33.07	43.	43.08	53.	53.08	63.	63.08	73.	73.07	83.	83.06	93.	93.05
4.	4.03	14.	14.04	24.	24.05	34.	34.07	44.	44.08	54.	54.08	64.	64.07	74.	74.07	84.	84.06	94.	94.05
5.	5.03	15.	15.04	25.	25.06	35.	35.07	45.	45.08	55.	55.08	65.	65.08	75.	75.07	85.	85.06	95.	95.04
6.	6.07	16.	16.04	26.	26.06	36.	36.07	46.	46.08	56.	56.08	66.	66.08	76.	76.07	86.	86.06	96.	96.04
7.	7.02	17.	17.04	27.	27.06	37.	37.07	47.	47.08	57.	57.08	67.	67.08	77.	77.07	87.	87.06	97.	97.04
8.	8.02	18.	18.04	28.	28.06	38.	38.07	48.	48.08	58.	58.08	68.	68.08	78.	78.07	88.	88.06	98.	98.04
9.	9.02	19.	19.05	29.	29.06	39.	39.07	49.	49.08	59.	59.08	69.	69.08	79.	79.07	89.	89.06	99.	99.04
10.	10.03	20.	20.05	30.	30.06	40.	40.07	50.	50.08	60.	60.08	70.	70.08	80.	80.07	90.	90.05	100.	100.03

QUALITY CONTROL COMMENTS

****** CURVE NOT STORED ON FILE

Attachment D

Analyzer ID 038284 MET- 1 DATE 5-16-78

Analysis for Hamilton Test Systems GAS C_3H_8 / N_2

Range 0- 100 ppm Sample Pressure 42" H_2O Flow 8

	CYLINDER #	CONCENTRATION	DVM 1	DVM 2	DVM 3	DVM 4
1	G-11829	99.39	99.35			
2	D-7393	81.64	81.5			
3	C-11849	77.52	77.55			
4	H-89480	49.55	49.5			
5	G-11835	37.14	36.95			
6	G-11857	24.37	24.4			
7	G-11873	18.92	18.9			
8	G-89472	12.76	12.7			
9						
10	S-31102	95.00	95.5			
11						
12						
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14						
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LR105

Environmental Protection Agency

Span Point Change Notice Procedure

This procedure is written for the Environmental Protection Agency, National Vehicle and Fuel Emissions Laboratory (NVFEL) internal use. The use of specific brand names by NVFEL in this procedure are for reference only and are not an endorsement of those products. This document may be used for guidance by other laboratories.

NVFEL Reference Number

205A

Implementation Approval

Original Procedure Authorized on 80-23-82

Revision Description

(1) 06-15-94 This procedure has been edited as described in EPCN #147. Page layout, grammatical, and spelling changes have been made, but there were no technical revisions to the procedure.

Table of Contents

Attachments

1. Purpose

The purpose of this procedure is to generate, verify, and document analyzer span points used by Vehicle Testing (VT), Heavy Duty (HD), and Evaluation and Development (E&D) gas analysis systems.

This procedure is used when a new analyzer curve is generated (TP 204) and/or when a span gas bottle is replaced (TP 502) or renamed (TP 105).

2. Test Article Description

An analyzer's “span point” is derived by projecting the Environmental Protection Agency (EPA)-named span gas concentration (derived from the Master Gas Analysis Site per TP 105) onto the active analyzer calibration curve to determine the corresponding deflection value for that particular analyzer/range.

The span point is used in emissions testing to set up the analyzer prior to analysis. A Span Point Change Notice Report (SPCN) is generated whenever an analyzer's span gas or calibration curve changes.

3. References

3.1 EPA Laboratory Safety Manual

3.2 Laboratory Computer System (LCS) GASCAL user's manual (PDW 60:03:04)
LCS CYSITE user's manual (PDW 60:03:16A)
LCS CNAP user's manual (PDW 60:03:05)
LCS Test Analysis Processor (TAP) user's manual (PDW 60:05:07B)

3.3 TP 204, Gas Analyzer Calibration Curve Generation

3.4 TP 502, Gas Cylinder Change

3.5 TP 105, Gas Naming

3.6 SPCN development file in Engineering Operation Division (EOD)

4. Required Equipment

4.1 Replacement span cylinder, if the span gas is being changed

4.2 Secondary standard cylinders, if a curve is being generated or checked. Refer to TP 204 for selection of proper standard cylinders.

5. Precautions

5.1 The technician must be familiar with the Laboratory Safety Manual, especially Chapters 2 through 6 dealing with the safe handling of compressed gases.

5.2 When possible, 24 hours advance notice should be given to Gas Analysis prior to changing a span bottle (see TP 502).

5.3 After a new span bottle is installed, it must be allowed to flow long enough to purge the plumbing lines and to allow the analyzer to stabilize before a span reading is taken.

5.4 Old span points must be crossed off the span point table of each analyzer site as soon as they are no longer valid.

5.5 New pending span points must be handwritten on the span point table at each analyzer site as soon as they are determined.

5.6 The name on the span cylinder must be less than one month old to be used.

6. Visual Inspection

Verify that the analyzer flow rate posted at the site remains constant both before and after a new span bottle is installed. Verify that the name on the span gas bottle is no more than one month old.

7. Test Article Preparation

7.1 If a span cylinder is being changed, perform TP 502.

7.2 If a new analyzer curve is being generated, perform TP 204.

8. Test Procedure

This procedure assumes a working knowledge of the LCS interactive computer system. Refer to the user's manual of the LCS programs referenced in this procedure for detailed instructions about terminal configuration, interactive commands, etc.

Sample runs of these programs are attached to this procedure.

The SPCN process consists of the following sections:

100 Replace/Rename Span Gas Cylinder -- to be performed when the working span gas is depleted or renamed.

200 Activate a New Analyzer Curve -- to be performed when a new analyzer curve is activated.

300 Span Point Validation -- performed by Data Validation after Section 100 or 200 has been performed.

400 Post Validated Span Point Tables, -- performed by Data Validation after Section 300 has been performed.

100 Replace/Rename Span Gas Cylinder

The current span gas cylinder sites in the National Vehicle and Fuel Emission Laboratory (NVFEL) are as follows:

COO1 - LDT (A001, A002, A003)
COO4 - LDT Diesel (A004, A016)
C021 - LDT SHEDs (A028, A029)
CO23 - LDT SHEDs (A027, A030)
C009 - HD Gasoline (A009)
C100 - HD Diesel (A101)
C200 - E&D (A202, A203, A015, A210, A014)

101 On the analyzer strip chart, record the site number; gas type; range number; current cylinder number; EPA tag concentration; EPA inventory number; and the posted span point.

102 Set up the analyzer for the new span bottle using one of the two conditions below:

If the old span cylinder can be used, zero the analyzer; then span gas flow it and obtain a stable and accurate span reading at all sites which share the gas.

Leaving the analyzer in the span mode, disconnect the old cylinder and connect the new one. Allow this gas to flow long enough to obtain a stable reading.

If the old span cylinder is not available or is suspected to be bad, zero the analyzer, flow the top secondary cylinder of the active curve for that analyzer, and obtain a stable and accurate reading at all sites which share the gas.

Then, as a check, flow the next secondary gas through the analyzer, which should respond within 0.5 deflections of the original deflection value of the active curve.

Leaving all the analyzers in the span mode, connect the new span cylinder and allow it to flow long enough to obtain stable readings at all sites which share the span gas.

Note: If the curve does not verify with the given tolerance, contact Calibration & Maintenance (C&M) and Gas Analysis to resolve the problem. If a span cylinder needs to be renamed, it must be done at the Master Gas Analysis Site.

103 Cross out the old span point for the replaced or renamed cylinder at all sites which share the gas. Enter the date and time, and initial next to the span point.

On the strip chart, mark the point of gas transfer of the old and new span points.

On LCS real-time supported sites, use LCS/TAP to take the analyzer readings.

If TAP is not available at a site, use a calibrated Fluke Model 8000A Digital Volt Meter (DVM) or equivalent, to take the analyzer readings. Write the DVM readings on the strip chart.

104 Run the LCS CYSITE program at an LCS production terminal. Press the BREAK key; then type “$RUN CYSITE” and press return.

105 Enter the current data as the program requests it. See Attachments A and B for sample runs of replaced and renamed gas cylinders.

If an analyzer range is inactive or out of service for some reason at a site which shares the span gas, enter "NONE" as that site's deflection reading. Be sure that the old span point for that range has been crossed out, dated, and initialed as per Step 103 above.

Write on the CYSITE printout the reason for entering "NONE."

106 The LCS CYSITE program will calculate a concentration for the new span cylinder. Check the percent difference (DIFF).

CYSITE will print a warning message if the percent difference between the calculated concentration and the EPA tag concentration is greater than ±1% of point.

If this occurs, check the new span bottle with secondaries per Step 102 (if this step has not been performed) and rerun CYSITE.

If the percent difference still exceeds 1%, contact C&M and Gas Analysis to resolve the difference as soon as possible. On the CYSITE printout, write the reason for the difference and notify the team leader before any testing begins.

107 Sign and date the LCS SPAN report as the operator. Enter the new calculated span point on each site's span point table.

Enter the date and time, and initial next to the new span point. The span point is now available for use on a pending basis, but it still must be certified by Data Validation as soon as possible.

108 If an error was made during a span gas change or during generation of the span point which was not detected until after CYSITE was run, notify the testing team leader so the problem can be rectified before any testing begins.

Verify that the old span point is crossed out so it will not be used accidentally. Do not submit anything to Data Validation until the problem has been rectified.

109 Collect the strip chart, the signed LCS CYSITE SPCN report, a copy of any old cylinder tags (pink), and the new cylinder tags (white).

If the span gas was renamed, copy the existing pink cylinder tag before and after the rename and label the copies "OLD" and "RENAMED." See Attachment F for sample cylinder tags.

Submit all these documents to EOD Data Validation for final review and approval.

200 Activate A New Analyzer Curve

201 Perform the curve generation (TP 204) and verify that the "calculated" concentration of the working gas, as named from the new curve, falls within ±1% of point of the EPA tag concentration.

This difference is known as "curve fit deviation" and can be found on the analyzer curve printout (see Attachment C for an example).

If the curve fit deviation is not within ±1% of point, the problem must be resolved by C&M and Gas Analysis.

202 After the curve and EPA tag concentration are determined to be valid and within tolerance, cross out the old span point at the analyzer site. Enter the date and time, and initial next to the span point.

203 Run the LCS CNAP program at an LCS production terminal in order to activate the pending calibration. Press the break key; then type "$RUN CNAP" then press return.

204 Enter the analyzer range calibration identification information as the program requests it. See Attachment D for a sample CNAP activation of a pending calibration for a new curve.

There is only one calibration number (YYMMDDHHMMSS) for a curve. It represents the exact time the data set was processed and is located in the upper left-hand corner of the pending analyzer printout.

205 After activating the calibration, CNAP will automatically print the LCS CNAP SPCN Report. Enter the new calculated span set point (as printed on the SPCN report) at the site's span point table.

Enter the date and time, and initial next to the span point. The span point is now available for use on a pending basis, but it still must be certified by the assigned technican as soon as possible.

206 If an error was made during the analyzer calibration or curve activation (or deactivation) which was not detected until after CNAP was run, notify the C&M Manager and the testing team leader so the problem can be rectified before any testing begins.

Verify that the old span point is crossed out so that it will not be used accidentally.

Do not submit anything to the assigned technican until the problem has been rectified.

207 Submit the signed LCS CNAP SPCN report, and a signed copy of the LCS GASCAL analyzer range calibration report to the assigned technican for final review and approval.

300 Span Point Validation

301 Verify that the LCS SPCN reports are signed and dated by the operator.

302 For SPCNs generated from new curves, check that the analyzer curve calculated concentration is within ±1% of point of the EPA tag concentration by examining the "curve fit deviation" on the LCS analyzer calibration report (see Attachment C for an example).

303 For SPCNs generated from new or renamed span gas cylinders:

Check that the deflection reading entered into the CYSITE agrees exactly with the LCS TAP or DVM readings.

On sites where LCS/TAP was not available, check the strip chart for 30 seconds of stable reading.

Check that any concentration differences greater than ±1% of point detected by CYSITE were resolved. Do not certify the span point table until this difference has been resolved.

Be sure the resolution is documented on the CYSITE printout.

Check that the cylinder IDs, gas type, analyzer range, and EPA tag concentration agree with the cylinder tags and were entered into LCS CYSITE correctly.

If an analyzer range is supposed to be inactive or out of service, check that "NONE" was entered as the deflection reading for that analyzer range (while using LCS CYSITE).

If a reading was entered, check with the operator to find out why and to resolve the problem.

304 Generate a span point table for each site listed in the LCS SPCN report using the LCS TAP program. See Attachment E for a sample TAP run to generate the span point table.

Check all table entries for the following:

If an analyzer range is inactive, check that no span point is printed in the site span point table for that analyzer range.

If a span point is printed, cross it out and contact C&M to "deactivate" that calibration curve using CNAP so it will not appear the next time the span point table is printed and posted by Data Validation.

If an analyzer range is out of service, cross out the associated span point printed in the span point table. This will insure that the span point will not be used until it is repaired.

Once the analyzer span point is repaired, a new span point table can be printed and posted at the site.

305 Stamp the span point table(s) with a Data Validation stamp.

If any error is found with the span point generation or with the pending handwritten value at the site, notify the team leader so the problem can be rectified.

400 Post Validated Span Point Tables

The validated span point tables must be updated at least once a week by Data Validation.

401 Take the certified span point tables to the analyzer sites. The table to be posted must be certified by EOD Data Validation to be considered official; it supersedes any other span point table generated by LCS.

402 Verify that the interim handwritten span point values agree with the new span point table.

403 After the new span point table is checked against the one posted at the site, trim the new copy to fit the bracket on the analyzer console.

404 Remove the old span point table and insert the new one.

405 Write the date/time of posting on the new span point tables. Return the extra copies and the old span point table to Data Validation.

9. Data Input

The data used to generate the LCS SPCN reports are input through the LCS programs CYSITE or CNAP. The data used to generate a pending analyzer calibration are input through the LCS GASCAL program.

10. Data Handling

10.1 The operator performing the SPCN procedure enters the data into the LCS computer via an LCS DEC Writer terminal.

10.2 LCS printouts are submitted to Data Validation for review.

11. Data Review and Validation

Data are reviewed and validated as part of the procedure. The technicians performing the procedure are responsible for addressing LCS warnings and for documenting any problems encountered.

Data Validation is responsible for officially accepting the new span point as outlined in Section 300, Part 8, and for posting updated span point tables at the analyzer sites.

12. Acceptance Criteria

12.1 For new analyzer curves, the percent difference between the calculated curve fit concentration and the EPA nominal tag concentration must be within ±1% of point for the new span set point to be accepted.

This is printed as the "curve fit deviation" by the LCS GASCAL program.

12.2 For new or renamed span bottles, the percent difference between the calculated curve fit concentration and the EPA tag concentration must be within ±1% of point for the new span set point to be accepted.

CYSITE prints a warning and will not update the data base if it is not within tolerance.

13. Quality Control Provisions

13.1 The site span point tables are dated and initialed whenever a span point is crossed out or whenever a new span point is entered.

13.2 Only Data Validation or senior technicians may update the site span point tables with certified LCS/TAP span point tables. Both are notified if any discrepancies are found.

13.3 Crossing out the old span points as soon as bottles are changed or curves are updated minimizes the possibility that old span points can be used accidentally.

13.4 Crossing out the span point for an analyzer range which is out of service minimizes the possibility that span point will be used accidentally.

14. Documentation

14.1 The following documentation is filed in Data Validation for SPCNs due to span gas cylinder changes:

the original signed LCS CYSITE SPCN report
the strip chart with DVM readings written on it
the old cylinder tag (pink) or copies of it
any new cylinder tag (white)

14.2 The following documentation is filed in Data Validation for SPCNs due to analyzer curve changes:

the original signed LCS CNAP SPCN report
a signed LCS CASCAL analyzer range calibration report
the old span point table that was replaced
C&M is responsible for keeping the curve strip charts.

Attachment A

TYPE BREAK KEY.

ATTACHMENT A -
SAMPLE CYSITE REPLACEMENT RUN

```
T.S.P. OPCOM Y PROD

??$RUN CYSITE


G A S   C Y L I N D E R   P R O G R A M

<> 12/09/91  08:14:47  CYSITE00 VERS:2.0-11/16/91

(ENTER <RETURN> TO USE DEFAULT VALUES IN PARENTHESES)
(ENTER "STOP" AT ANY TIME TO TERMINATE THE PROGRAM.



REPLACE OR RENAME TRANSACTION? (TTTTTTT):REP
  CYLINDER SITE? (CNNN): C021
    NEW CYLINDER ID? (WNNNNN): W005B1
     OLD CYLINDER ID? (WNNNNN):W00415
        OPERATOR ID? (IIIII):17281
          GAS TYPE? (TTTT):C3H8
            ANALYZER RANGE? (RR):16
             (WAITING FOR EFS CURVE ACCESS)
              EPA TAG CONCENTRATION? (XXXXXXXXXX): 92.28
                 SITE:    A029
                 ENTER    XX.X
                 DEFL?   92.3
                 CONC:    92.293
                 DIFF:     0.01 %
                 ARE DATA ENTRIES CORRECT? (Y/N):Y
                  (EFS UPDATE BEGINNING)

    SPCN REPORT       EFFECTIVE D/T: 12/09/91  08:16:56
    ----------        ---------------------------------
    ANALYZER SITE   :     A029
    EQUIPMENT TYPE  :     HCAN
    LAB STD RANGE   :     16
    SPAN SET POINT  :     92.3

    ANALYZER ID     :     037910
    ANALYZER USAGE  :     BAGA
    ANALYZER CAL NO.:     810825091030

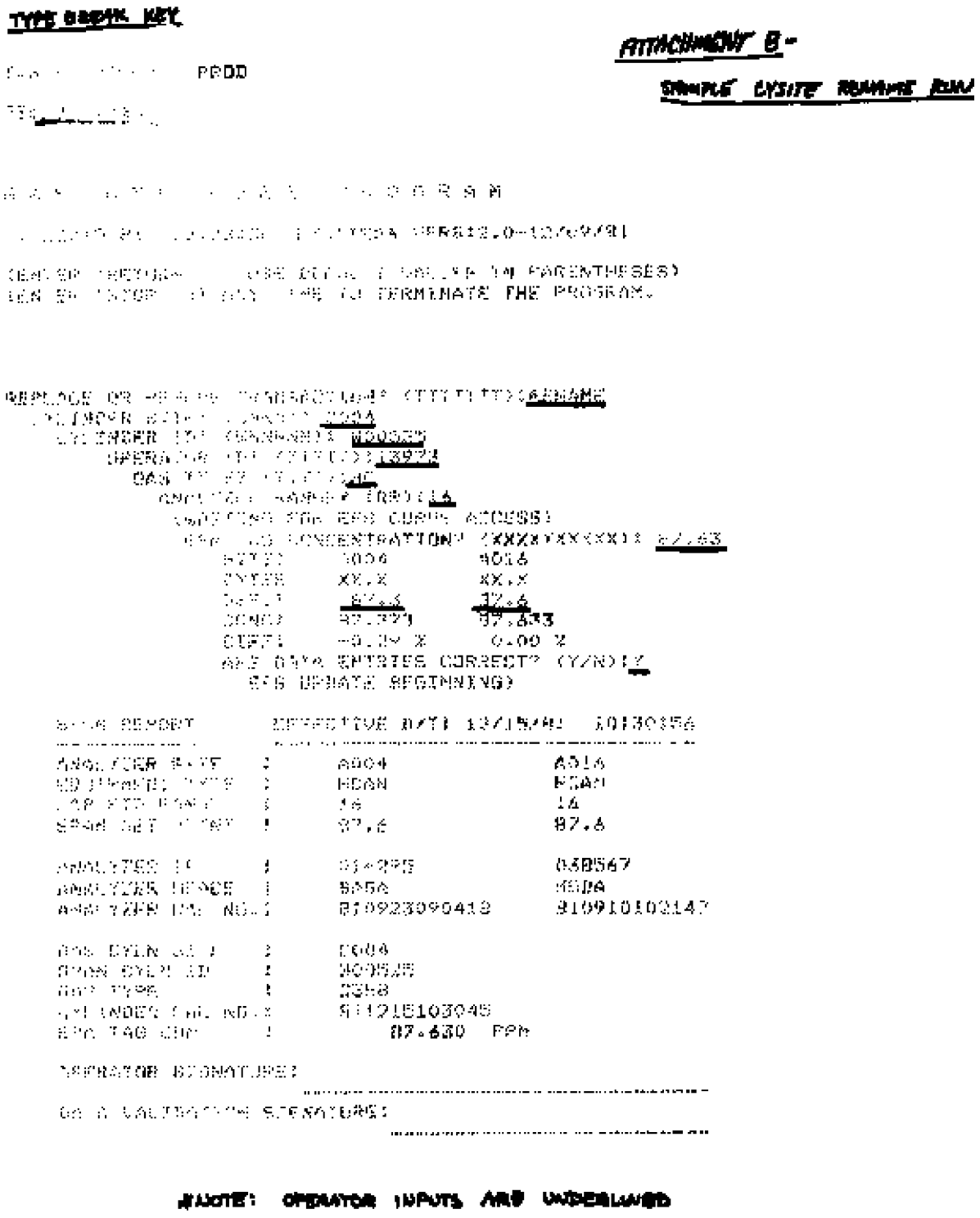
    GAS CYLN SITE   :     C021
    SPAN CYLN ID    :     W005B1
    GAS TYPE        :     C3H8
    CYLINDER CAL NO.:     811209091446
    EPA TAG CONC    :         92.280  PPM

    OPERATOR SIGNATURE:  ____________________________

    DATA VALIDATION SIGNATURE:  ____________________________
```

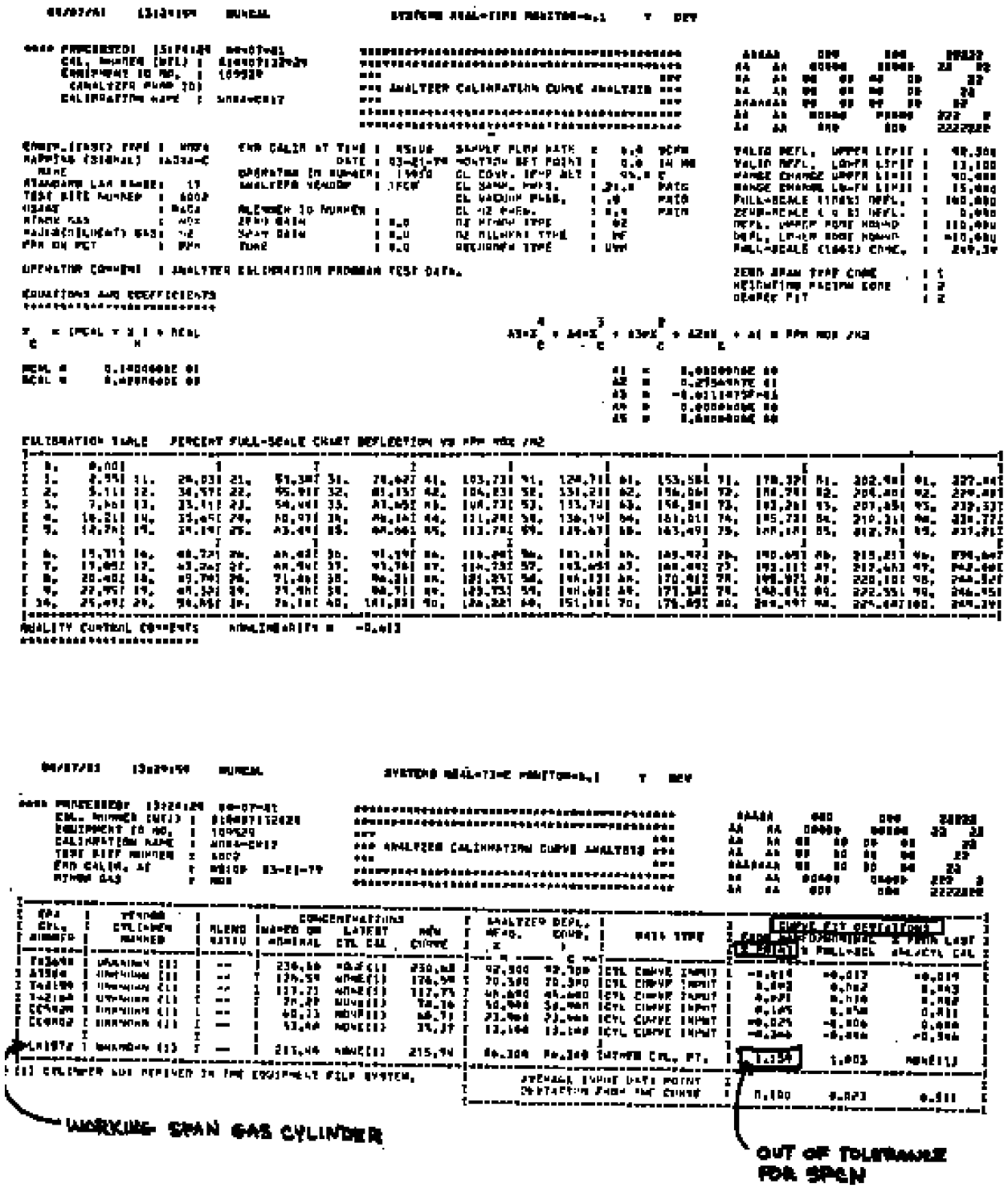
NOTE: OPERATOR INPUTS ARE UNDERLINED.

Attachment B



Attachment C

ATTACHMENT C – LCS GASCAL REPORT



WORKING SPAN GAS CYLINDER

OUT OF TOLERANCE FOR SPAN

Attachment D

TYPE CRAK KEY

ATTACHMENT D –
CNAP Calibration curve activation

```
T.G.P. OPCOM X PROD

?T$RUN CNAP


C A L I B R A T I O N   N A M E   A C T I V A T I O N   P R O G R A M

<> 12/03/81  10:46:20  CNAP  06 VER$:2.0-11/03/81

COMMAND> ACTIVATE
EQ ID NO>086985
CALIBRATION NAME>HCAN-CR15
CALIBATION NO.>810909092625
    12/03/81  10:46:53 OLD CALIBRATION DEACTIVATED
    12/03/81  10:47:01 NEW CALIBRATION ACTIVATED
    EFS ACCESS FOR ANALYZER AND CYLINDER CURVES BEGUN.

    SPCN REPORT       EFFECTIVE D/T: 12/03/81  10:47:01
    ----------        ------------------------------
    ANALYZER SITE   :     A009
    EQUIPMENT TYPE  :     HCAN
    LAB STD RANGE   :     15
    SPAN SET POINT  :     92.7

    ANALYZER ID'    :     086985
    ANALYZER USAGE  :     BAGA
    ANALYZER CAL NO.:     810909092625

    GAS CYLN SITE   :     C009
    SPAN CYLN ID    :     W00056
    GAS TYPE        :     C3H8
    CYLINDER CAL NO.:     811203090211
    EPA TAG CONC    :          46.250  PPM

    OPERATOR SIGNATURE:
                       ------------------------------
    DATA VALIDATION SIGNATURE:
                              ------------------------

COMMAND> STOP

<> 12/03/81  10:49:57  CNAP  06 TERMINATED NORMALLY
```

NOTE: operator inputs are underlined.

Attachment E

ATTACHMENT E –

TAP span point table printout

TYPE BREAK KEY

T.G.P. OPCON X DEV

??RUN TAP

T E S T   A N A L Y S I S   P R O G R A M

<> 12/15/81 10:04:11 TAP   00 VERS:1.2-12/05/81
>>ANALYZER SITE? A004

10:04:14 WAITING FOR CALIBS TO LOAD

12/18/81 10:04:53 A004 SPAN SETPOINTS
APPROVAL: INITIALS, DATE, TIME
HB . 12/15/81 08:50

	HC		NOX		CO2		LCO		HCO	CH4
----	14	81.5	15	89.8	22	82.0	17	9?.4		
----	15	95.2	16	91.6	23	87.4	18	??.4		
----	16	87.6	17	91.1						
----	HC		NOX		CO2		LCO		HCO	CH4

10:05:16 * NO RTP ASSIGNED TO THE SITE   ----

<> 12/18/81 10:05:18 TAP , 00 TERMINATED NORMALLY

Note: operator inputs are underlined.

Attachment F

ATTACHMENT F – SAMPLE EPA CYLINDER TAGS

old span #66-2

EPA GAS CYLINDER (White)
INVENTORY RECORD
(LEFT JUSTIFY AS APPLICABLE)

NOMINAL CONC.	CONC. UNITS	MINOR COMP.	DILUENT TYPE
200	PPM	CO2	AIR

DATE RECEIVED	VENDOR CODE	CYLINDER NO.
++		A-9399

EPA INVENTORY CONTROL NO.	MULTIBLEND CONTROL NO.	EQUIPMENT TYPE
400581		

DATE NAMED	NAMED CONC.	OPERATOR EPA I.D. NO.
120881	9218	11687

DATE IN SERVICE	GAS CAT.	SITE* CODE	LSR*	USAGE*
120881		A029		

*APPLIES TO SPAN GASES ONLY

DATE SHIPPED FROM EPA LAB	EPA INVENTORY CONTROL NO.	SHIPPER EPA I.D.
++		

NEW CYLINDER

EPA GAS CYLINDER (Pink)
INVENTORY RECORD
(LEFT JUSTIFY AS APPLICABLE)

NOMINAL CONC.	CONC. UNITS	MINOR COMP.	DILUENT TYPE
8420	PPM	CO2	AIR

DATE RECEIVED	VENDOR CODE	CYLINDER NO.
++		A-4266

EPA INVENTORY CONTROL NO.	MULTIBLEND CONTROL NO.	EQUIPMENT TYPE
160415		

DATE NAMED	NAMED CONC.	OPERATOR EPA I.D. NO.
062781	8619	36693

DATE IN SERVICE	GAS CAT.	SITE* CODE	LSR*	USAGE*
062781		5001		SPAN

*APPLIES TO SPAN GASES ONLY

DATE SHIPPED FROM EPA LAB	EPA INVENTORY CONTROL NO.	SHIPPER EPA I.D.
++		

OLD CYLINDER

Attachment G

Span Point Change Notice				
New Analyzer Curve				
Analyzer Site Number				
Gas Type				
Range Number				
SPAN Cylinder Number				
EPA Tag Concentration	Y3			
Posted SPAN Set Point				
SPAN Chart Defl. Reading from Curve	X0			
Upper Chart Deflection Bracket	X2			
Lower Chart Deflection Bracket	X1			
X2 - X1	^2X			
Calc. Curve Fit Concentration	Y0			
Upper Concentration Bracket	Y2			
Lower Concentration Bracket	Y1			
Y2 - Y1	^2Y			
% Difference = $\frac{Y0 - Y3}{Y3}$ x 100% (Must be < ± 1.0%)				
New Curve derived SPAN Set Point $X3 = X1 ({}^2X / {}^2Y) (Y3 - Y1)$	X3			
Curve Verification SPAN Change				
Date of Curve Verification				
Old SPAN Set Point				
New SPAN Set Point	X0			

Calculated By: _____

Validated By: _____

Date / Time Effective: _____

X2 = the next higher chart deflection to X0 found on the curve calibration tab
X1 = the next lower chart deflection to X0 found on the curve calibration tab
Y2 = the next higher concentration to Y0 found on the curve calibration tab
Y1 = the next lower concentration to Y0 found on the curve calibration tab

Form 204-01: 05-20-94

Environmental Protection Agency

Gas Correlation Procedure

This procedure is written for the Environmental Protection Agency, National Vehicle and Fuel Emissions Laboratory (NVFEL) internal use. The use of specific brand names by NVFEL in this procedure are for reference only and are not an endorsement of those products. This document may be used for guidance by other laboratories.

NVFEL Reference Number

403A

Implementation Approval

Original Procedure Authorized 11-07-78

Revision Description

(1) 06-15-94 This procedure has been edited as described in EPCN #147. Page layout, grammatical, and spelling changes have been made, but there were no technical revisions to the procedure.

Table of Contents

Attachments

1. Purpose

The purpose of this procedure is to quantify and assure the traceability of primary and secondary gas blends to National Institute of Standards and Technology (NIST) standards and to each other.

As part of the correlation procedure, those gases to be used as Environmental Protection Agency (EPA) laboratory secondary standards are "named."

All correlated primary and secondary standard gases may be used as calibration points in "TP 105, Gas Naming." All named secondary standards may be used throughout the Engineering Operations Division (EOD) as calibration standards.

2. Test Article Description

A correlation is performed for each of the following gas blends:

CO/N_2, CO_2/N_2, NO/N_2, Propane/N_2, Propane/Air, Methane/Air, O_2/N_2, H_2/He, H_2/N_2 and $HC/O_2/N_2$

The correlation's incorporate all available NIST cylinders, EPA-blended gravimetric (designated as primary standards), and all cylinders to be used as laboratory secondary standards.

3. References

3.1 Instruction manuals for the instruments on the Master Analysis System

3.2 "EPA Laboratory Safety Manual"

3.3 "Federal Register," Vol. 43, Section 86.114

3.4 Proceedings of the "EPA/Industry Quality Control Symposium on Gas Standards - Management and Traceability Practices," July 27, 1977, particularly the "Quality Assurance Paper on Calibration Gas Management," Don Paulsell, March, 1976

3.5 Memo from Don Paulsell, March 23, 1978, Subject: "Confidence Limits on Gas Naming Procedure"

4. Required Equipment

4.1 The following components contained in the Master Analysis System A251:

4.1.1 Beckman Model 400 Flame Ionization Detector (FID) for measuring hydrocarbons

4.1.2 Gow-Mac Series 550 Thermal Conductivity Gas Chromatograph for analyzing H_2/He and H_2/N_2

4.1.3 Horiba Model AIA-23A and AIA-23AS infrared analyzers with different cell lengths for analyzing CO and CO_2

4.1.4 Beckman Model 951 NO/NOx Analyzer

4.1.5 Bendix 8205 Methane Analyzer

4.1.6 Taylor Servomex Type 0A.137 Oxygen Analyzer

4.1.7 Fluke 8000A Digital Multimeter

4.1.8 Hewlett-Packard 7132A Strip Chart Recorder, used for methane correlation

4.2 Regulators and sample lines. Sample lines should be Teflon, covered with braided stainless steel and with stainless steel fittings. All regulators must be dual stage.

NOx regulators must be stainless steel, connection size CGA 660, with Teflon gaskets.

Brass regulators are used for all other gases, connection size CGA 590 for air, CGA 580 for nitrogen, and CGA 350 for all other gases.

4.3 All available NIST and gravimetric cylinders and cylinders designated to be used as EPA secondary standards of the gas blend to be correlated

4.4 Zero gases - hydrocarbon-free air or 99.9% nitrogen as required

4.5 Form LB105, Gas Analysis Data Worksheet

4.6 Form AA-601, Exhaust Gas Analysis Calibration Data Form

4.7 Form LB403, Correlation Data Sheet

5. Precautions

5.1 Technicians must be familiar with the "EPA Laboratory Safety Manual" sections on cylinder safety and the safe handling of test gases.

5.2 All cylinders and equipment (regulators, lines, instruments, etc.) must be checked for leakage, damage, and cleanliness.

5.3 Labels on all cylinders to be used must be carefully read to determine their contents.

6. Visual Inspection

Not applicable. All visual inspections are included as part of the test preparation and test procedure.

7. Test Article Preparation

7.1 Assemble all available NIST and gravimetric cylinders and cylinders to be used as laboratory secondary standards.

7.1.1 Organize the cylinders in order from the highest concentration to the lowest.

7.1.2 Partition the cylinders into a series corresponding to the analyzer instrument ranges.

Each range should include all cylinders whose nominal concentrations fall between the high end of the range and not below 10% of that range (e.g., if the range is 0-20,000 ppm, the lowest should be approximately 2,000 ppm).

For best results, there should be at least 8 cylinders within each range, and each range should overlap the next by as many cylinders as possible.

7.2 The ambient room and cylinder temperatures must be between 68 °F and 86 °F while the analysis is run. Be sure to allow at least 24 hours for cylinders that have just been received to adjust to this temperature range.

7.3 Make sure the cylinders are properly secured. Attach suitable pressure regulators to all cylinders. Check for leaks with leak detector solution.

7.4 Analytical instruments used for test gas calibration should always remain in a standby mode to insure maximum accuracy and stability.

If the instrument has been shut off, it must be warmed up according to the analyzer operations manual.

8. Test Procedure

All calibration points must be stable readings taken from the digital volt meter (DVM). A stable reading must be determined by the operator as that reading with as little fluctuation as possible. The operator must allow sufficient time for the reading to stabilize.

If a reading fluctuates between two points, the operator estimates the average reading (e.g., 90.1-90.2 = 90.15). Fluctuations greater than 0.2 units should be noted on Form LB105.

If a methane correlation is being performed, the strip chart is used to verify instrument stability. Note on the strip chart all corresponding readings taken from the DVM.

100 Calibrate the analyzer.

101 ZERO: Switch the analyzer to zero and allow zero gas to flow through it. Adjust the pressure if necessary, check the flow rate, and select the proper range. These must remain constant during the analysis.

Adjust the zero potentiometer to read between ±0.1 unit of zero.

102 SPAN: Attach the analyzer input line to the highest concentration standard gas in the range being analyzed.

Switch the analyzer to span, wait for the reading to stabilize, and adjust the span potentiometer so that the reading falls between 92 and 99 percent of full scale.

103 ZERO: Switch the analyzer to zero.

If the reading does not fall between ±0.1 unit of zero, adjust the zero potentiometer and repeat the span-zero until the zero point remains within tolerance.

200 Analyze all gases to be used as EPA standards.

201 Switch the analyzer to span. Allow the reading to stabilize and record it on Form LB105.

202 Disconnect the input line from the span gas and connect it to the next lower concentration standard cylinder. Record the DVM reading after it stabilizes.

Repeat this process for the remaining standard gases in the range being tested in order from the highest concentration to the lowest.

203 If the low CO analyzer is being used (0-100 ppm), check the zero point after each standard cylinder is analyzed. Check the span point after half the standard bottles have been analyzed. It must be within ±0.1 unit of the first span reading.

If the zero and span points do not fall within tolerance, repeat the analysis until they do.

204 After the lowest concentration standard is recorded, reconnect the highest concentration standard and note the DVM reading once it stabilizes.

If the reading is not within ±0.1 unit of the first span reading, zero the analyzer and repeat Steps 200-206 until the two span points agree within this tolerance limit.

205 Switch the analyzer to zero and obtain a stable reading.

If the reading is not within ±0.1 unit of zero, calibrate the analyzer and analyze the standard gases (Steps 100 - 206).

206 Repeat Steps 100 - 206 for all of the analyzer ranges.

9. Data Input

9.1 The operator completes Form LB105 for each range analyzed, giving the analyzer ID, usage code, data, who the analysis is for, the type of gas and diluent, range, pressure, and flow rates.

The cylinders are listed by number, nominal or known concentration, and by the digital voltmeter reading. Four columns are provided for the DVM readings in the event of rerun curves.

9.2 The operator completes Form AA-601 for each range analyzed using the information recorded on Form LB105.

9.2.1 Lines 1-3, Instrument Identification, are completed using the codes given on the back of the form.

9.2.2 Line 4, Limits, is completed. The deflection limits define the upper and lower limits of valid deflection readings on the DVM. The range change limits define the upper and lower deflection readings on the DVM that signal the need for a range change to computers on real-time systems.

9.2.3 On Line 5, Operator's Comments, EPA is identified as the recipient of the results and the type of standards used are given (NIST, grav, secondary).

9.2.4 Columns 1-11 of Line 7 are completed using the codes on the back of the form as follows:

Cols. 1-1, zero-span type - Always “01” (“no software zero and span”).

Cols. 4-5, curve form - “01” if the curve is to be forced through zero.
“02” if the curve is to have a finite intercept.

Col. 8, degree of fit - “2” if the chemiluminescence, FID, methane, or oxygen analyzer was used.
“4” if the NDIR analyzer was used.

Col. 11, weight factor - always “2,” which minimizes percent of point deviations.

Cols. 16-51 - are applicable only if a blender was used.

9.2.5 Lines 8-9 are applicable only is a blender was used.

9.2.6 Lines 10-29 are concerned with the cylinders involved in the analysis:

Cols. 1-12 - The cylinder numbers are listed.
Col. 14 - applicable only if a blender was used.
Col. 16 - “X” if the cylinder concentration is “known.”
Col. 18 - “X” if the cylinder concentration is “to be named.”
Cols. 20-32 - applicable only if a blender was used.
Cols. 34-44 - each cylinder known or nominal concentration is listed.

Col. 46 - "X" if the cylinder is to be used as a calibration data point. All such cylinders must have a known concentration value.

Cols. 48-55 - the DVM readings taken from Form LB105 are recorded to two decimal places.

Note: Typically, gravimetric standards are used as "calibration data points" and secondary standards and NIST cylinders are marked "to be named."

9.3 The operator completes two Standard Gas Correlation Sheets, LB403; one for NIST and gravimetric cylinders, the other for secondaries; extracting the appropriate information from the Analyzer Curve Analysis printout.

9.3.1 In Column 1, the cylinder numbers of the primary and secondary standards are listed on separate sheets, in order from the highest concentration to the lowest (top to bottom).

9.3.2 In Column 2, the nominal concentration is listed beside the appropriate cylinder number.

If the secondaries have no previous EPA-correlated concentration, this column is left blank.

9.3.3 In Columns 3-14, the calculated concentrations of all the cylinders analyzed are listed by range. These concentrations are found on the Analyzer Curve Analysis printouts.

9.3.4 The average concentration is calculated for each cylinder by totaling the different calculated concentrations of the ranges and dividing the sum by that number of ranges. This average is recorded in Column 15.

If a secondary cylinder has not been previously correlated, the average becomes the "name" of that cylinder.

9.3.5 The percent of deviation is calculated by the following formula:

$$\% \text{ DEV} = \frac{(\text{Avg-Nom})}{\text{Nom}} \times 100\%$$

This quotient is recorded to three decimal places in Column 16.

If the secondary cylinders have no previous EPA-correlated concentration, this column is left blank.

9.3.6 The percent range is calculated to show the relative spread in the data for each cylinder using the following formula:

$$\% \text{ RANGE} = \frac{(\text{Max-Min})}{\text{AVG}} \times 100\%$$

where Max = highest reading of the range spread, and

MIN = lowest reading of the range spread

10. Data Handling

10.1 A Data Processing Request Form is completed and Form AA-601 is turned in with it for processing.

10.2 The processed data sheet, Analyzer Calibration Curve Analysis, is obtained from Computer Operations.

11. Data Review and Validation

11.1 The technician examines the Analyzer Calibration Curve Analysis for each analyzer range and determines the validity of the curves.

11.1.1 A valid curve is determined by noting the figures in the column under "curve fit deviation" marked "% point." All figures in this column must be less than ±0.5% for the curve to be valid.

11.1.2 No inflection points are allowed in the curve. These are flagged in the printout and indicate problems either with the analyzer or with the gas.

11.1.3 The percent of non-linearity routinely found under the data table of the printout must be less than 10. Non-linearity greater than 10 is flagged in the Quality Control Comments of the printout. In such cases, the section chief should be notified for further action.

11.1.4 The plus and minus signs of deviations (±) must be randomly distributed throughout the curve.

If three or more of the same sign appear clustered at the center or endpoint, the section chief should be notified for further action.

If the curve is not valid, the input data must be checked for accuracy, corrected, and reprocessed if necessary.

If the input data are found to be accurate but the curve is invalid, a problem with the cylinder contents or analyzer is indicated. The curve must be rerun to verify this. Once the problem is verified, the source must be determined and rectified.

If the problem is with inaccurate cylinders, these cylinders may be removed from the curve by eliminating the X's in Columns 16 and 46 of Form AA-601, adding an X in Column 18, and reprocessing the data. The abnormal cylinders should either be reblended or returned to the vendor.

If the curve is valid, the data may be used in the correlation procedure.

11.2 The technician examines the correlation sheets and determines the validity of the correlation.

11.2.1 The percent of deviation between the calculated and nominal concentrations must be no more than ±0.5%.

11.2.2 The percent of range-to-range deviation for each cylinder must be no more than 0.5%.

If either of the above figures is out of tolerance, the section chief should be notified for further action.

After the correlation has been determined to be valid, the correlated cylinders may now be used as standards in TP 105, the Gas Naming procedure, and secondaries may be used for calibrations in the Engineering Operations Division.

12. Acceptance Criteria

12.1 All zero and span points must fall between ±0.1 unit of the original readings.

12.2 All curves must be valid according to the criteria set in Step 11.1 before the correlation can be made.

12.3 The primary standard correlation must be valid according to the criteria set in Step 11.2.

The secondary standard correlation must be valid according to the criteria set in Step 11.2.2 for those cylinders not previously correlated.

If a secondary cylinder has been previously correlated, any subsequent correlations must be valid according to the criteria set in Step 11.2.

13. Quality Control Provisions

13.1 All analytical instruments must be properly warmed up prior to use.

13.2 Any abnormal cylinders must be removed from the analysis and replaced by cylinders that meet the acceptance criteria.

14. Documentation

14.1 Forms LB105, AA-601, LB403, and the Analyzer Calibration Curve Analysis are filed separately in the Master Analysis Room.

14.1.1 Forms LB105 and AA-601 are filed chronologically, according to the gas type. Methane correlation strip charts are attached to and filed with Form LB105.

14.1.2 The Analyzer Calibration Curve Analysis printouts are filed chronologically, according to the gas type, in individual notebooks for primary and secondary standards.

14.1.3 Form LB403 is filed according to the gas type.

14.2 A black sticker type label is affixed to each correlated secondary cylinder giving the cylinder number, the gas blend, and the average concentration taken from Form LB403.

14.3 A summary report on the correlation project is sent to all laboratory branch chiefs and to the Quality Assurance Manager.

Attachment A

Analyzer ID C-38254 MET- 1 DATE 5-5-78

Analysis for C of H correlation - Groningues GAS C3H8 / N2

Range 0- 150, / 100, / 50, / 250 Sample Pressure 42" a.o flow 8 scfh

	CYLINDER #	CONCENTRATION	0-1500 DVR 1	0-1000 DVR 2	0-200 DVR 3	0-250 DVR 4
1	D 1425	485.11	95.73	X	X	X
2	G 11537	1236.20	79.5	X	X	X
3	G-11555	917.55	59.1	98.3	X	X
4	G 11561	760.77	50.1	83.5	X	X
5	D-7395	606.23	39.0	65.0	X	X
6	D-7472	492.10	31.65	5275	96.25	X
7	G-11846	384.61	24.8	41.3	75.4	X
8	D-7473	369.66	23.8	39.7	72.3	X
9	G 11844	232.82	15.0	25.0	45.6	98.15
10	D-7405	151.82	9.75	16.2	29.6	63.7
11	D-7455	95.569	X	10.25	18.7	40.4
12	D 7424	74.434	X	X	14.6	31.5
13	D-7431	61.969	X	X	12.15	26.25
14	D-7448	47.319	X	X	9.3	20.1
15	D-7451	37.549	X	X	X	15.9
16	D 7453	23.564	X		X	99
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LM105

Attachment B

Analyzer ID 038284 UNIT 1 DATE 5-15-78

Analysis for Lab Calibration Secondaries GAS CH_4 / N_2

Range D= 0-100, 0-200, 0-500, 0-2500 Sample Pressure 42" H_2O flow 8 scfh

	CYLINDER #	CONCENTRATION	0-1000 DVM-1	0-1000 DVM-2	0-500 DVM-3	0-2500 DVM-4
1	13027	1154.0	72.35	X	X	X
2	A 1865	792.0	49.1	81.8	X	X
3	AM124	614.0	38.65	64.45	X	X
4	A 4001	398.9	25.35	42.2	77.15	X
5	A 1953	304.8	19.3	32.2	56.85	X
6	A 565	226.7	13.5	22.6	41.2	85.9
7	A 363	180.8	11.5	19.2	35.1	75.6	
8	A 2520	156.0	9.5	15.9	29.1	62.75
9	A 4659	127.7	X	13.5	24.9	53.4
10	A 5272	94.6	X	10.0	18.3	39.5
11	A 6166	76.9	X	X	15.15	32.8
12	A 5976	62.0	X	X	12.3	26.6
13	A 5424	45.7	X	X	9.05	19.7
14	A 2613	35.9	X	X	X	15.9
15	A 1310	30.1	X	X	X	13.0
16	A 2765	23.4	X	X	X	10.1
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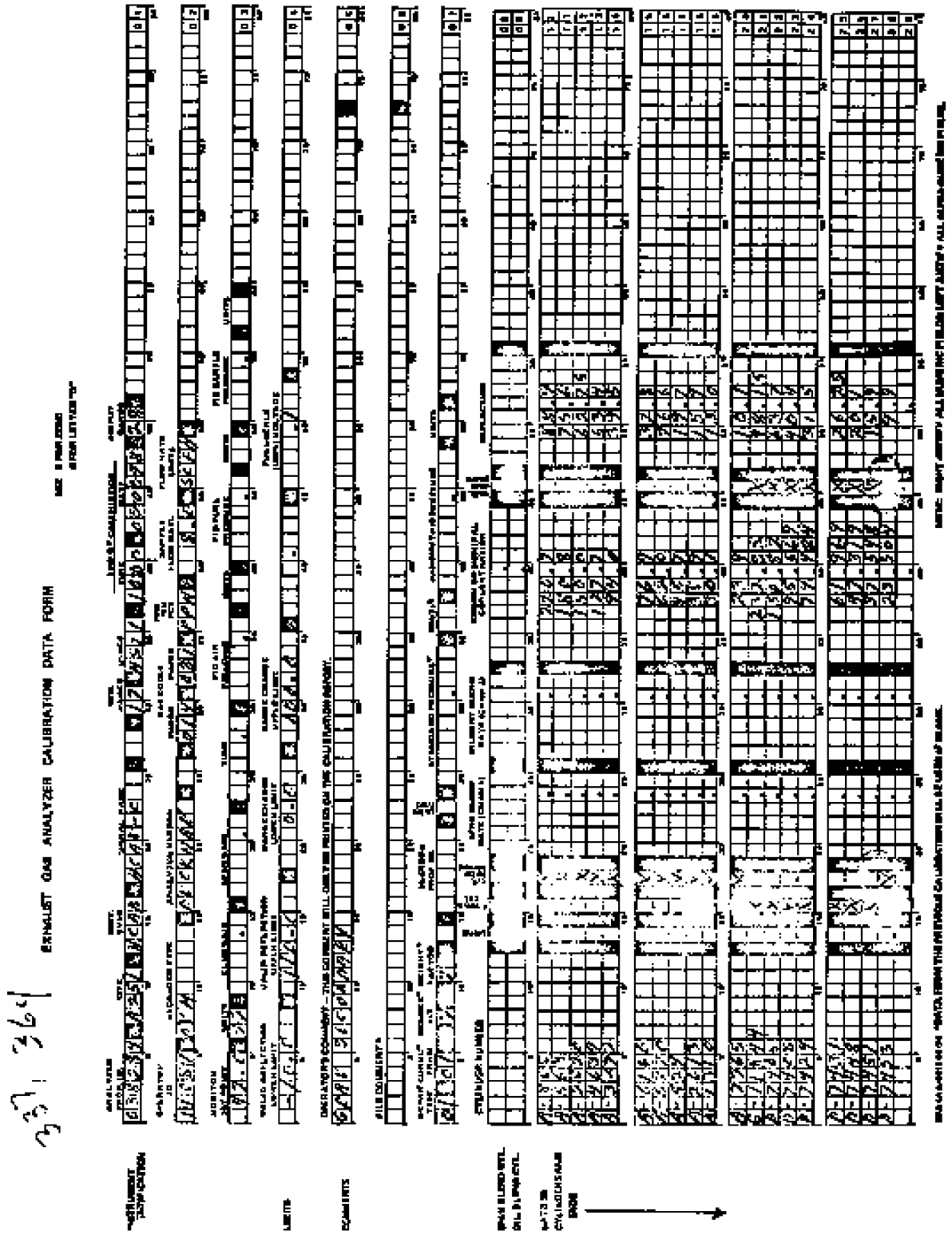
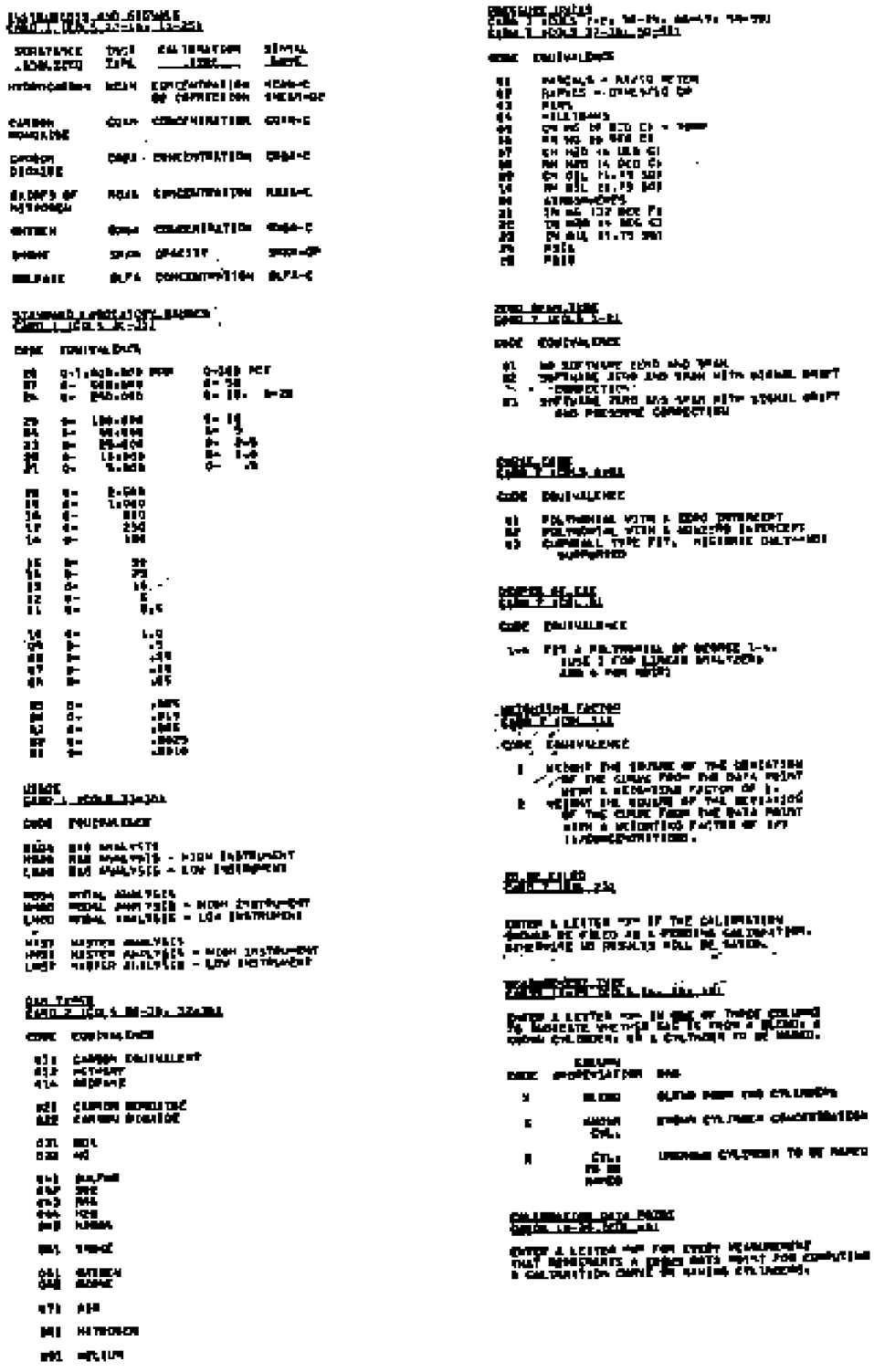
LB105

Attachment C



Attachment D



Attachment E

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**** PROCESSED: 1311842N  05-30-7.

**************************************************
**************************************************
***                                            ***
*** ANALYZER CALIBRATION CURVE ANALYSIS        ***
***                                            ***
**************************************************
**************************************************
```

END CALIB AT TIME : 10: 0	ANALYZER VENDOR	: BECKMAN	SAMPLE FLOW RATE : 0.0 SCFM	VALID DEFL. UPPER LIMIT :	110.000
CALIBRATION DATE : -- 8-79	INST- PRINT NAME	: HCAN	MONITOR SET POINT : 42.0 IN H2O	VALID DEFL. LOWER LIMIT :	-10.000
TEST SITE NUMBER : 4251	EPA DECAL ID NO	: 13-796	ZERO GAIN SETTING : 0.0	RANGE CHANGE UPPER LIMIT :	100.000
GAS ANALYZED : PROPANE	SIGNAL LEAD	: MAIN-C	SPAN GAIN SETTING : 0.0	RANGE CHANGE LOWER LIMIT :	0.0
DILUENT GAS : NITROGEN	HARDWARE RANGE	:	TUNE READING : 0.0	FULL-SCALE (100%) DEFL. :	100.000
CONCENTRATION UNITS : PPM	USAGE	: HST1	FID AIR PRESSURE : 0.0	FULL-SCALE (100%) VOLTAGE :	0.108
STANDARD LAB RANGE : 17	CALIB GAS SOURCE	:	FID FUEL PRESSURE : 0.0	FULL-SCALE (100%) CONC. :	237.3
OPERATOR ID NO : 11089	BLENDER DECAL ID	:	FID SAMP PRESSURE : 0.0	RECORDER TYPE	1044

FILE COMMENT :
OPERATOR COMMENT : GRAY SECONDARY

CYLINDER NUMBER	BLENDER METER READINGS SPAN	BLENDER METER READINGS DILUENT	BLEND RATIO	CONCENTRATIONS CYLINDER (BLENDED)	CONCENTRATIONS CALCULATED	ANALYZER SIGNAL MEASURED %	ANALYZER SIGNAL CORRECTED %	CALIBRATION DATA POINT	CURVE FIT DEVIATIONS % POINT	CURVE FIT DEVIATIONS % FULL-SCALE	CURVE FIT DEVIATIONS % FROM LAST CALIBRATION
	%			0.0		M	C				
		%		0.0							
14-364	0.0	0.0	0.0	226.70	210.79	88.900	88.900		0.0	0.0	0.0
14-543	0.0	0.0	0.0	180.90	179.09	75.600	75.600		0.0	0.0	0.0
14-2620	0.0	0.0	0.0	150.00	144.53	62.750	62.750		0.0	0.0	0.0
14-4139	0.0	0.0	0.0	127.70	126.32	53.400	53.400		0.0	0.0	0.0
14-1277	0.0	0.0	0.0	94.00	93.35	39.500	39.500		0.0	0.0	0.0
14-4156	0.0	0.0	0.0	76.90	77.48	32.800	32.800		0.0	0.0	0.0
14-4770	0.0	0.0	0.0	62.00	62.81	26.600	26.600		0.0	0.0	0.0
14-526	0.0	0.0	0.0	45.70	46.49	19.700	19.700		0.0	0.0	0.0
14-2617	0.0	0.0	0.0	35.90	37.52	15.900	15.900		0.0	0.0	0.0
14-1310	0.0	0.0	0.0	30.10	30.67	13.000	13.000		0.0	0.0	0.0
14-1765	0.0	0.0	0.0	23.90	23.92	10.100	10.100		0.0	0.0	0.0
10-11844	0.0	0.0	0.0	232.42	232.46	98.150	98.150	X	0.019	0.018	0.0
10-7405	0.0	0.0	0.0	150.42	150.76	63.700	63.700	X	-0.024	-0.015	0.0
10-7408	0.0	0.0	0.0	95.57	95.48	40.400	40.400	X	-0.092	-0.037	0.0
10-7424	0.0	0.0	0.0	74.42	74.40	31.500	31.500	X	-0.026	-0.009	0.0
10-7431	0.0	0.0	0.0	61.97	61.96	26.250	26.250	X	0.019	0.005	0.0
10-7448	0.0	0.0	0.0	47.32	47.44	20.100	20.100	X	0.255	0.051	0.0
10-7451	0.0	0.0	0.0	37.55	37.52	15.900	15.900	X	-0.084	-0.013	0.0
10-7453	0.0	0.0	0.0	23.50	23.35	9.900	9.900		0.0	0.0.	0.0
								AVERAGE DEVIATION	0.074	0.021	0.0

NONLINEARITY = 0.2 PERCENT

Attachment F

```
**** PROCESSED: 1112129  05-30-7                ****************************************          AAA     22222   5555555     11
                                                ****************************************         AAAAA   2222222  5555555    111
                                                ***                                  ***        AA   AA       22  55         1 11
                                                *** ANALYZER CALIBRATION CURVE ANALYSIS ***     AAAAAAA     222   555555       11
                                                ***                                  ***        AAAAAAA   222          55      11
                                                ****************************************        AA   AA  2222222  5555555      11
                                                ****************************************        AA   AA  2222222   55555     11111

END CALIB AT TIME  :  10: 0     ANALYZER VENDOR   : BECKMAN    ZERO SPAN TYPE    :    L
CALIBRATION DATE   : 5- 8-78    INSTRUMENT NAME   :  HCAH      CURVE FORM        :    L
TEST SITE NUMBER   :  A251      EPA LOCAL ID NO   : 032244     DEGREE FIT        :    2
GAS ANALYZED       : PROPANE    SIGNAL LEAD       : CAN-C      WEIGHTING FACTOR  :    2
FULL-SCALE CONC    :    237.3   MANT ARE RANGE    :
CONCENTRATION UNIT : PPM        USAGE             :  RST1
STANDARD LAB RANGE :   17       OPERATOR ID NO    : 11049

EQUATIONS AND COEFFICIENTS
**************************
                                                                 4        3        2
         X  = X  I                                       (A5*X  + A4*X  + A3*X  + A2*X  + A1) =   PPM PROPANE/NITROGEN
          C    N                                              C        C        C        C

                                                                     A1  =  0.0
   CALIBRATION P(HSPO) =  0.0                                        A2  =  0.2357017E+01
                                                                     A3  =  0.1580499E-03
                                                                     A4  =  0.0
                                                                     A5  =  0.0
```

CALIBRATION TABLE ____ PERCENT FULL-SCALE CHART DEFLECTION VS PPM PROPANE/NITROGEN

0.	0.0																		
1.	2.36	11.	25.45	21.	49.57	31.	73.22	41.	96.90	51.	120.62	61.	144.37	71.	168.14	81.	191.96	91.	215.80
2.	4.71	12.	29.31	22.	51.93	32.	75.59	42.	99.27	52.	122.99	62.	146.74	72.	170.52	82.	194.34	92.	218.18
3.	7.07	13.	30.67	23.	54.30	33.	77.95	43.	101.64	53.	125.37	63.	149.12	73.	172.90	83.	196.72	93.	220.57
4.	9.43	14.	33.03	24.	56.66	34.	80.32	44.	104.01	54.	127.74	64.	151.50	74.	175.28	84.	199.10	94.	222.96
5.	11.79	15.	35.39	25.	59.02	35.	82.69	45.	106.39	55.	130.11	65.	153.87	75.	177.67	85.	201.49	95.	225.34
6.	14.15	16.	37.75	26.	61.38	36.	85.06	46.	108.76	56.	132.49	66.	156.25	76.	180.05	86.	203.87	96.	227.73
7.	16.51	17.	40.11	27.	63.74	37.	87.43	47.	111.13	57.	134.86	67.	158.63	77.	182.43	87.	206.26	97.	230.12
8.	18.87	18.	42.48	28.	66.12	38.	89.79	48.	113.50	58.	137.24	68.	161.01	78.	184.81	88.	208.64	98.	232.51
9.	21.23	19.	44.84	29.	68.49	39.	92.16	49.	115.87	59.	139.61	69.	163.39	79.	187.19	89.	211.03	99.	234.89
10.	23.59	20.	47.20	30.	70.85	40.	94.53	50.	118.25	60.	141.99	70.	165.77	80.	189.57	90.	213.41	100.	237.26

QUALITY CONTROL COMMENTS
****

****** CURVE NOT STORED ON FILE

Attachment G

ANALYSIS OF CYLINDERS TO BE NAMED

CYLINDER NUMBER	NOMINAL CONCENTRATION (NC)	LINEAR FIT SIGNAL			LINEAR FIT CALC. CONC. (LC)	CURVE FIT CALC. CONC. (CC)	CONCENTRATION RATIOS	
		LOW	HIGH	CYLINDER			NC/CC	LC/CC
A-868	226.70	63.700	99.150	86.400	210.80	210.79	1.07549	1.00007
A-549	190.80	63.700	99.150	75.600	179.15	179.04	1.06953	1.00029
A-2520	150.00	0.400	67.700	62.750	149.57	148.53	1.00993	1.00028
A-4679	127.70	0.400	63.700	53.400	126.40	126.32	1.01096	1.00064
A-3272	94.0000	41.500	40.400	39.403	93.4307	93.3648	1.00698	1.00068
A-6165	76.9000	41.500	40.400	32.403	77.5124	77.4802	0.99251	1.00042
A-5770	62.0000	20.250	31.500	26.403	62.7493	62.8685	0.98713	0.99965
A-524	45.7000	15.400	29.100	19.700	46.3984	46.4966	0.98291	0.99772
A-2613	35.9000	15.400	29.100	15.944	37.5440	37.5165	0.95691	1.00084
A-1310	30.1000	15.900	20.100	13.003	30.8014	30.6474	0.98148	1.00435
A-2765	23.9000	15.400	20.100	10.100	24.0947	23.8220	1.00327	1.01177
D-7453	23.5049	15.900	20.100	9.403	23.5496	23.3500	1.00660	1.01026

Attachment H